

Accelerated Shelf Life Testing of *Chocomix* Using Critical Moisture Content Approach

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(Received: June 16, 2017; Accepted: March 23, 2018)

Abstract

Chocomix is a chocolate powder drink made from cocoa beans by local farmers in Nglanggeran Village. This study aims to investigate the shelf life of Chocomix using the Accelerated Shelf Life Testing (ASLT) method, particularly the critical moisture content approach. The result of the study shows that the critical moisture content of Chocomix is 4.01% (db). The MSI curve of Chocomix in a temperature of 28°C forms an isothermal sigmoid curve type II with two curves in the range of a_w 0.24 and a_w 0.68. The equation obtained from the MSI curve is $y = 86.584X^3 - 91.893X^2 + 28.818X - 0.470$. The shelf life of Chocomix stored at a temperature of 28°C in a metalized film with a thickness of 51.89 μm is 16.4 months.

Keywords: chocolate powder; critical moisture content; moisture sorption isotherm; shelf life

How to Cite This Article: Nurhayati, R., Pratiwi, R., Anandito, B.K., Herawati, E.R.N., and Angwar, M., (2018), Accelerated Shelf Life Testing of Chocomix Using Critical Moisture Content Approach, *Reaktor*, 18(2), 63-70, <http://dx.doi.org/10.14710/reaktor.18.2.63-70>.

INTRODUCTION

Cocoa is a type of plantation commodities that grows well in the tropics like Indonesia. One of the cocoa producing regions in Indonesia is Gunungkidul Regency, Special Region of Yogyakarta. According to the statistical data of Gunungkidul Regency (2015), Gunungkidul produced 254.65 tons of cocoa in 2014 with 110.65 tons alone produced by Patuk subdistrict. Patuk is the most productive cocoa growing region in Gunungkidul. One of the ways to increase the value added of cocoa is to process it into various kinds of processed cocoa products (Rahman, 2015). *Griya Cokelat Nglanggeran* has been processing cocoa into

various products (Herawati *et al.*, 2017), one of which is a chocolate powder drink called *Chocomix*. Nonetheless, the shelf life of this product has not been confirmed yet.

The Institute of Food Science and Technology defines food shelf life as the period between the manufacture and the purchase of a food product, during which time the product is in a state of satisfactory conditions in terms of appearance, taste, smell, texture, and nutritional value (Arpah, 2001). However small a food industry is, it must determine the shelf life of its food products, thereby offering consumers a food safety guarantee (Harris and Fadli, 2014). In addition,

there are other regulations on expiration date as enacted in Law No. 8 Year 2012 on Food and Government Regulation No. 69 Year 1999 on Food Labeling and Advertisement, requiring that all food industries to mention the expiration date of their product on its packaging. By providing this information, the manufacturers do not only benefit the customers in that they will know the time limit to purchase or use the products, but also help the distributors or retailers manage their stocks and check the product quality.

Chocolate powder drinks are susceptible to damage due to the adsorption of moisture from the surrounding environment. Rosniati and Dume (2010) affirm that chocolate powder is very hygroscopic and if it is not well-packed, the fat crystals in the powder will easily melt during storage, leading to clumping and discoloration. Further, Mustafidah and Widjanarko (2015) elaborate that the level of clumping is one of the parameters to define the quality of powdered food products. Consumers tend to reject a powder drink when it is clumped.

Kusnandar *et al.* (2010) assert that one of the methods used to determine the shelf life of a product is the Accelerated Shelf Life Testing (ASLT). In ASLT, the shelf life of a product is determined by storing the product under controlled conditions that increase the rate of degradation, either under higher temperature or elevated moisture. The acceleration method can be carried out in a shorter time with good accuracy. Since this study aims to predict the shelf life of a chocolate powder drink, the ASLT approach employed in this study was the critical moisture content approach, i.e. an approach used for predicting the shelf life of foods which are easily damaged due to moisture adsorption.

RESEARCH METHOD

Material

The sample used in this study was a chocolate powder drink produced by *Griya Cokelat Nglanggeran*, called *Chocomix*. This product is packed in a metalized film. The salts used to balance the material in various a_w levels were lithium chloride (LiCl), magnesium chloride ($MgCl_2$), potassium carbonate (K_2CO_3), magnesium chloride [$Mg(NO_3)_2$], sodium nitrite ($NaNO_2$), sodium chloride (NaCl), potassium chloride (KCl), and potassium sulfate (K_2SO_4).

Physicochemical Properties

The physical property testings of the product included: a color testing using Konika Minolta CR-400 Chromameter with the Hunter $L^*a^*b^*$ system (Misnawi, 2011). Meanwhile, the chemical property testings involved such aspects as: moisture content using the termogravimetric method (AOAC, 1970), ash content by the drying method (Sudarmadji *et al.*, 2010), fat content using the Soxhlet method (Sudarmadji *et al.*, 2010), protein content by the Kjeldahl method (Apriyantono *et al.*, 1989), and carbohydrate content by difference (Winarno, 2004).

Determination of Initial Moisture Content (M_i)

The initial moisture content was determined by carrying out the oven-drying method for 6 hours at a temperature of 105°C.

Determination of Critical Moisture Content (M_c)

The critical moisture content as an indicator of deterioration can be determined by storing the sample without package at room temperature. In every two hours, from 0 to 10 hours, the panelists carried out both a preference test and a moisture content test. The obtained data from both tests were then plotted into a linear regression equation. The critical moisture content was obtained by plotting a score of 3 (somewhat clumping) into the linear equation of the regression results from the correlation curve between moisture content and preference score. The resulted moisture content was called as the critical moisture content (M_c).

Determination of Equilibrium Moisture Content (M_e)

The equilibrium moisture content (M_e) was obtained by placing some samples in a jar filled with saturated salt with various levels of a_w . Two grams of sample was stored in an aluminum cup. This cup was stored in a jar that was closed tightly, and then kept at room temperature (28°C). Changes in the sample's weight were observed at certain time intervals until constant weight was obtained. After reaching the constant weight, the moisture content (db) of each sample was analyzed. This moisture content was called the equilibrium moisture content (Kusuma *et al.*, 2015). A graphic then was used to depict both the M_e and the a_w . This particular graphic was called the MSI curve with a_w as the x -axis and M_e as the y -axis. From this curve, an MSI curve equation could then be formulated based on the third degree polynomial as follows:

$$M = A a_w^3 + B a_w^2 + C a_w + D \quad (1)$$

Where A, B, C and D are equation constant.

The general form of the Guggenheim – Anderson – de Boer (GAB) equation was used to calculate both the a_w of the material and the critical moisture content (M_c). The obtained M_i , M_c , and a_w values were plotted in a curve with the a_w as the x -axis and moisture content as the y -axis. The linear regression equation was used to calculate the value of the equilibrium moisture content (M_e). The general form of the GAB equation can be mathematically written as follows:

$$\frac{M}{M_o} = \frac{K.C.a_w}{(1-K.a_w)(1-K.a_w+C.K.a_w)} \quad (2)$$

Determination of Package's Water Vapor Permeability

The packaging permeability is determined using the Permatran Mocon-W 3//31 at RH 90% and a temperature of 37.8°C. This device showed the value of Water Vapor Transmission Rate (WVTR, g/m²/day). The permeability constant (k/x) is calculated by

dividing the WVTR by the result of the multiplication of pure water vapor pressure (P_o) at a temperature of 37.8°C with the RH value.

Shelf Life Estimation

After obtaining the initial moisture content of the material (M_i), critical moisture content (M_c), equilibrium moisture content (M_e), and permeability of packaging material to water vapor (k/x), the researchers could then determine the shelf life of the material based on the sorption isotherm curve approach using the Labuza equation (1984) as shown as follows:

$$\theta = \frac{\ln\left(\frac{M_e - M_i}{M_e - M_c}\right)}{\frac{k(A)}{x(W_s)} \frac{P_o}{b}} \quad (3)$$

Where,

- M_e = equilibrium moisture content at ambient temperature and RH (g moisture/100 g dry solid), based on straight-line equation on MSI curve.
- M_o = Initial moisture content of product (g moisture/100g)
- M_c = Critical moisture content (g moisture/100 g dry solid)
- k/x = Permeability of package (g moisture/m².day mmHg)
- A = Surface area of package (m²)
- W_s = Product weight in package (g)
- P_o = Pure water vapor pressure at experimental temperature (mmHg)
- b = Slope of MSI curve in storage operational area
- θ = Shelf life (day)
(Labuza, 1984)

RESULTS AND DISCUSSION

Physicochemical Characteristics of *Chocomix*

The L^* , a^* , and b^* values of *Chocomix* are 62.19, 9.77, and 11.93 respectively. Misnawi (2011) asserts that the measurement of L^* value with a scale of 0 – 100 shows a brightness level which is in the range of black to white. Positive a^* indicates a redness level in a scale of 0 – 60, and negative a^* shows a greenness level in the scale of 0-(-60). Meanwhile, positive b^* is yellow in a scale of 0 – 60, and negative b^* is blue in a scale of 0-(-60). An L^* value of 62.19 indicates that the color of *Chocomix* is nearly white, while a positive a^* value of 9.77 results in a more reddish color. In addition, a positive b^* value of 11.93 suggests that the *Chocomix* color is yellow. Similar results are also reported by Vissotto *et al.*, (2010). They find that a chocolate powder drink has $L^*a^*b^*$ values of 57.76, 7.80, and 10.87 respectively.

A °Hue value is the dominant wavelength that is used to determine the material or product color (Winarno, 2008). Misnawi (2011) states that a °Hue value can be calculated using the L^* , a^* , b^* with the following formulation:

$$^{\circ}\text{Hue} = \tan^{-1} \left(\frac{b}{a} \right) \quad (4)$$

The calculation results in a °Hue value of 50.66. Pramesta *et al.* (2012) affirms that a °Hue value in a range of 18 – 54 indicates a red color.

Chocomix is made from chocolate powder, mixed with both milk powder and sugar powder. The chemical characteristics of *Chocomix* is presented in Table 1.

Table 1. Chemical characteristics of *Chocomix*

Parameter	Value (%)
Water content	1.62
Ash content	2.61
Fat content	6.24
Protein content	7.31
Carbohydrate by difference	82.22

Initial Moisture Content

Initial moisture content is one of the factors affecting the shelf life of a product. The findings of this study indicate that the initial moisture content of *Chocomix* is 1.64% (db). This moisture content is measured on a dry basis. The moisture content of *Chocomix* is categorized as “low”. Winarno (2008) states that a high moisture content in a food product will shorten the shelf life of the product, and vice versa, the lower the moisture content is, the longer the shelf life of the product will be. In addition, initial moisture content is related to the critical moisture content of a product (Kusnandar, 2006). The greater the difference between the initial moisture content and the critical moisture content of food product is, the longer the shelf life of the product will be.

Critical Moisture Content

Critical moisture content is one of the product’s acceptance quality limits by consumers. According to Mustafidah and Widjanarko (2015) consumers may refuse a powder drink when clumping has occurred. It is organoleptically assumed as a critical moisture content in which the *Chocomix* chocolate drink texture is no longer acceptable to consumers.

The graph of the moisture content relationship during observation with the preference score of the *Chocomix* chocolate powder drink can be seen in Figure 1. The graph shows that the higher the value of the moisture content is, the lower the preference score will be. This is because the longer it is stored, the greater the water vapor adsorption will be and the more visible the clumping is. The regression equation used is $y = -0.8572x + 6.5796$ with R^2 of 0.9824. From the regression equation, the obtained critical moisture content is 4.01% (db).

Equilibrium Moisture Content

Equilibrium moisture content of food is obtained when the water vapor pressure of the material is in equilibrium with its environment, meaning that the product weight does not change (Fellows, 1990). Equilibrium moisture content in food products is used to determine and describe the isothermal sorption curve of the product. The curve is used to obtain information

on moisture transfer during the adsorption or the desorption process (Pavinee, 1998). A Moist Sorption Isotherm (MSI) curve is a curve that connects moisture content with a water activity of materials at a certain temperature (Labuza, 1984).

The determination of the equilibrium moisture content of *Chocomix* is done by conditioning the sample in saturated salt solutions with different RH levels at a temperature of 28°C. The sample will experience water vapour adsorption from the environment and vice versa. This water vapour transfer occurs due to differences in RH environments and products, where water vapors will move from a higher RH to a lower RH. This will occur until equilibrium between the moisture content of the *Chocomix* and the storage environment is reached. The equilibrium is indicated by a constant sample weight. Based on the research carried out, the measurement results of equilibrium moisture content on *Chocomix* at various levels of a_w value can be seen in Table 2.

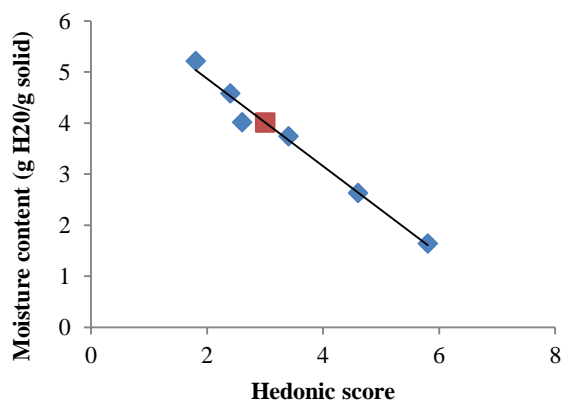


Figure 1. Correlation graphic between moisture content and hedonic score of *Chocomix* chocolate beverage powder

Table 2. Equilibrium moisture content of *Chocomix* instant chocolate beverage powder at various a_w levels

Salt	Water activity (a_w)	Equilibrium moisture content (% db)
LiCl	0,11	1,03
MgCl ₂	0,33	1,75
K ₂ CO ₃	0,44	2,17
Mg(NO ₃) ₂	0,52	2,46
NaNO ₂	0,64	3,55
NaCl	0,75	5,26
KCl	0,84	9,49
K ₂ SO ₄	0,97	20,26

The data in Table 2 show that the higher the water activity (a_w) is, the higher the equilibrium moisture content of *Chocomix* will be. This is due to the water activity (a_w) in the storage environment affecting the amount of water adsorption. The higher the water activity (a_w) is, the greater the number of water vapors adsorbed by the material to reach the equilibrium will be. Based on Table 2, the water activity data (a_w) and

the equilibrium moisture content can be plotted using a third degree polynomial to get a MSI curve (Figure 2.).

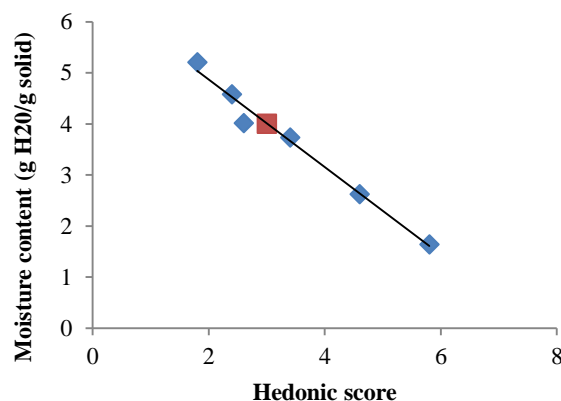


Figure 2. Moisture sorption isotherm curve (MSI) of *Chocomix* instant chocolate beverage powder

As depicted in Figure 2, the MSI curve of the *Chocomix* forms an MSI curve type II that follows the S (Sigmoid) pattern. In line with this, Labuza (1984) states that an MSI curve type II is a typical curve for dried food and grain. This sigmoid shape is formed by the effect of capillarity and the interaction between the material surface and water molecules (Adawiyah, 2006). On the MSI curve, *Chocomix* has 2 curves, which are in the range of 0.24 and 0.68 a_w .

According to Labuza (1984), the water adsorption of dried food was cumulatively influenced by the physicochemical effect so that two curves seem to appear. The limit between the primary and secondary bound water ranges from 0.2 to 0.4 a_w , while the limit between the secondary bound water and free water is 0.6-0.7 a_w . Bound primary water has tightly bound water, which forms hydrates; the water is relatively difficult to be removed or evaporated. Bound secondary water is weakly bound water because it is adsorbed on the surface of macromolecular colloids such as protein, pectin, starch, and cellulose. Water in this form is still free and can be crystallized in the freezing process. Meanwhile, free water is contained in the inter-cell space and intergranular of the material. Free water can fasten the process of food damage through microbiological, chemical, enzymatic processes, and even through destruction by insects (Septianingrum, 2008). The third degree polynomial equation obtained from the MSI curve of the *Chocomix* chocolate drink is $y = 86.584x^3 - 91.893x^2 + 28.818x - 0.4697$; with $R^2 = 0,9907$

There are 23 equations that can explain the relationship between the water content and the a_w of foods (McLaughlin and Magee, 1998). One of the internationally recognized isotherm sorption equation models is the Guggenheim, Anderson, and de Boer (GAB) model. This model can illustrate the isotherm sorption of food in the wider range of $0.05 < a_w < 0.9$ (Spiess and Wolf, 1987). Moreira *et al.* (2010) add that the GAB model can represent a_w 0.0 to 0.94. Furthermore, based on the testing on grains, the model

shows a high validity. The GAB equation model is derived from the GAB curve using the second degree polynomial equation. This equation shows the relationship between the a_w value of salt and the a_w value of the equilibrium moisture content. The GAB equation in general can be mathematically expressed as follows:

$$\frac{M}{M_o} = \frac{K C a_w}{(1-K.a_w)(1-K.a_w+C.K.a_w)} \quad (5)$$

With:

K, C = Constant

a_w = Water activity

M = Moisture content (kg moisture/kg dry solid)

M_o = Monolayer Moisture content (kg moisture/kg dry solid)

The general form of the GAB equation is used to measure the initial moisture content (Mi) and the critical water content (Mc) of *Chocomix*. After the a_w values from both are obtained, the initial moisture content (Mi), critical water content (Mc), and the a_w value of each are plotted to obtain a regression equation that can be used to determine the equilibrium water content (Me). The data on the determination of the GAB curve of the *Chocomix* can be seen in Table 3. Then, the data are plotted to form the GAB curve with the second degree polynomial equation shown in Figure 3.

Table 3. Water activity and a_w /Me of *Chocomix* chocolate beverage powder

Salt	Water activity (a_w)	a_w /Me (db)
LiCl	0,11	0,11
MgCl ₂	0,33	0,19
K ₂ CO ₃	0,44	0,20
Mg(NO ₃) ₂	0,52	0,21
NaNO ₂	0,64	0,18
NaCl	0,75	0,14
KCl	0,84	0,09
K ₂ SO ₄	0,97	0,05

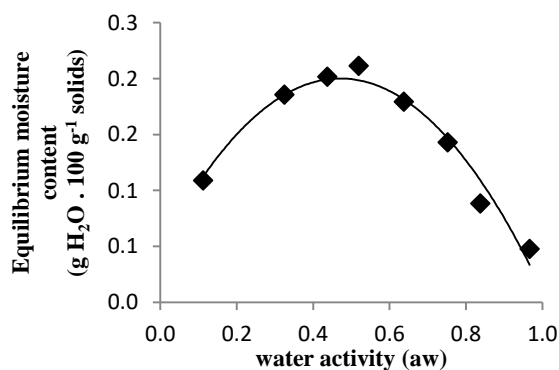


Figure 3. GAB curve equation *Chocomix* chocolate beverage powder

Based on the GAB equation curve of *Chocomix* in Figure 3, the equation is presented as follows:

$$y = -0.6784x^2 + 0.6395x + 0.0494 ; R^2 = 0.9660$$

The constants in this equation can be used to calculate the values of K, C, and M_o (K=0.99, C=15.13, and M_o=1.36). The mathematical analysis of the GAB equation shows that an equation will produce a good description of the sorption isotherm event with a sigmoid type if the K and C constants are in the range of 0.24<K≤1 and 5.67≤C≤∞ (Purwanti, 2012). In line with this, Medeiros *et al.* (2006) state that if the value of C is greater (>) than 10, the MSI curve is included in type II. According to Adawiyah and Soekarto (2010), the value of K equals to a multilayer water energy constant (above monolayer water). Quirijins *et al.* (2005) state that when the value of K equals (=) to 1, the molecule that passes through the monolayer has the same properties as pure water. The value of C describes the bond strength of the water molecule with the primary bond of the surface layer of the material. The greater the C value is, the stronger the bound of the water molecules contained in the monolayer and in binding the surface layer of the material will be. The C value resulted from the GAB equation of *Chocomix* is 15.13, so that the C value and the MSI curve obtained are appropriate. The M_o value describes the water content in the monolayer of the material. The water content in this monolayer is very important in determining the physical and chemical stability of the dried material (Aini *et al.*, 2014). Furthermore, the values of K, C, and M_o that have been obtained from the calculation are covered in the general form of the GAB equation which is presented as follows:

$$\frac{M}{1.36} = \frac{14.92 a_w}{1 + 12.95a_w - 13.73a_w^2} \quad (6)$$

or

$$M = \frac{20.24 a_w}{1 + 12.95a_w - 13.73a_w^2} \quad (7)$$

From the above GAB equation, the a_w value of the initial material (Mi) is 0.24, and the critical a_w value (Mc) is 0.68. The initial material of *Chocomix* is in the range of 0.2-0.4 a_w which is the limit between primary and secondary bound water. This means that *Chocomix* is in a safe condition because the a_w value is close to the primary bound water which is difficult to use in microbial activities or other chemical damages. Whereas the critical a_w value of the *Chocomix* chocolate drink is 0.68; this is in the range of 0.6-0.7 a_w that becomes the boundary between the secondary bound and free water. The closer *Chocomix* to the free water is, the easier it will be to experience damages. Hence, the critical a_w value (0.68) is the a_w value of *Chocomix* that has been damaged and has caused the decline, causing the drink to be rejected by the consumers.

After obtaining the a_w values from both, the data on the initial moisture content (Mi), critical water content (Mc), and a_w values of each are plotted and the regression equation is obtained ($y = 5.6049x - 0.2755$). This equation is used to determine the equilibrium moisture content (Me). According to Labuza (1984), equilibrium moisture content (Me) is derived from the water content in the work area of isotherm, which is represented by a straight line equation on the MSI curve, which passes through Mi and Mc. The storage

condition of *Chocomix* is assumed at a temperature 28°C and RH 75%. Therefore, based on the regression equation, the equilibrium water content (Me) of *Chocomix* is 4.38% (db).

Permeability of Packaging Materials

The packaging permeability factor needs to be considered because it affects the shelf life of a product. *Chocomix* is packed in a metalized film with a thickness of 51.89 μm . Fellows and Axtell (2002) state that a metalized film contains a thin layer of aluminum metal. The permeability of packaging material to water vapor is shown by the amount of water vapor that can pass through a package per day in certain atmospheric conditions and can be stated in gram $\text{H}_2\text{O}/\text{day}/\text{m}^2$ (Supriyadi, 1993). The thickness of the metalized film is 51.89 μm and the WVTR value of it is 0.71 g $\text{H}_2\text{O}/\text{m}^2/\text{day}$. Furthermore, the WVTR value is converted to k/x value. The value of the packaging permeability (k/x) was calculated by dividing the WVTR value with the results of the pure water vapor pressure (P_0) multiplication at the testing temperature of 37.8°C with the RH values (Kusnandar *et al.*, 2010). The pure water vapor pressure at 37.8°C is 49.17 mmHg so that the value of k/x is 0.02 $\text{gH}_2\text{O}/\text{m}^2\text{Day}.\text{mmHg}$.

The results of the moisture vapor permeability test are not much different from those of previous research by Kusnandar *et al.* (2010), which state that the two metalized films tested have a permeability of 0.0136 and 0.0180 g $\text{H}_2\text{O}/\text{m}^2\text{Day}.\text{mmHg}$. A metalized film has lower permeability than others, one of which is propylene. The lower the water vapour permeability of the packaging is, the greater the packaging ability to hold the water vapour will be, leading to a longer shelf life (Priyanto *et al.*, 2005).

Shelf Life Prediction

In the equation for shelf life prediction, there are supporting parameters other than those described above, namely solid weight per pack (W_s), pure water vapor pressure at a temperature of 28°C, packaging area (A), and slope (b). The slope of the isotherm curve (b) is determined by plotting the initial water content (M_i) and critical water (M_c) data into a straight line equation. The straight line equation that passes through M_i and M_c is shown in Figure 4. Thus, a slope of 0.05 is obtained.

The metalized film used to pack *Chocomix* has a size of 14.2 cm x 8.4 cm so that the packaging area is 0.01 m^2 . The weight of the *Chocomix* is 25 g per pack, so that the solid weight is 24.59 g. The saturated vapor pressure at a temperature of 28°C is 28.35 mmHg. Furthermore, related to the shelf life prediction, all data that have been obtained are covered in the Labuza equation (1984). The result of the shelf life prediction of *Chocomix* chocolate drink is shown in Table 4.

Based on the Table 4, the estimated shelf life of *Chocomix* chocolate drink wrapped using a metalized film with a thickness of 51.89 μm at RH 75% and a temperature of 28°C is 16.39 months. The shelf life of

a product is affected by the package. According to Priyanto *et al.* (2005), a metalized film has a packaging permeability toward low water vapor. Therefore, the material has a longer shelf life because of the greater ability of the package to hold water vapor.

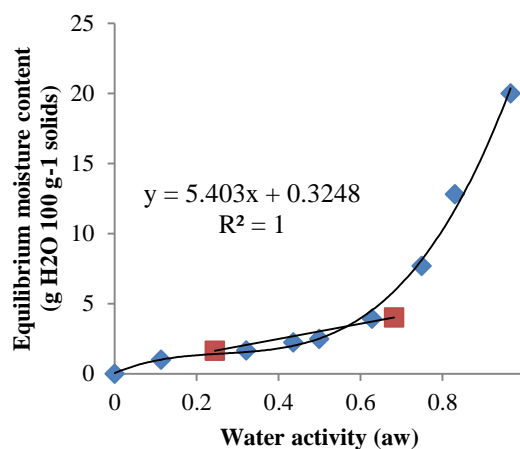


Figure 4. Straight-line equation passed through M_i and M_c

Table 4. The data of *chocomix* shelf life prediction

Parameter	Value
Initial moisture content (M_i , $\text{gH}_2\text{O}/100\text{g solid}$)	1,64
Critical moisture content (M_c , $\text{gH}_2\text{O}/100\text{g solid}$)	4,01
Equilibrium moisture content (M_e , $\text{gH}_2\text{O}/100\text{g solid}$)	4,38
Permeability package (k/x , $\text{gH}_2\text{O}/\text{m}^2\text{ day}.\text{mmHg}^2$)	0,02
Weight of solid material (W_s , g)	24,59
Surface area of package (A , m^2)	0,01
Pure water vapor pressure (P_0 , mmHg)	28,35
Slope (b)	0,05
Shelf life (θ , day)	491,76
Shelf life (θ , months)	16,39

CONCLUSION

Chocomix chocolate powder drink has a critical water content of 4.01% (db). From the results of the study, the Moist Sorption Isotherm (MSI) curve forms an isothermal sigmoid curve type II with 2 curves in the range of 0.24 and 0.68 a_w . *Chocomix*, of which the shelf life is predicted using the ASLT method, particularly the critical water content approach, and that is packed using a metalized film with a thickness of 51.89 μm at a temperature of 28°C and RH 75%, has a shelf life of 16.39 months.

ACKNOWLEDGEMENTS

We are gratefully thanks to Bank Indonesia Yogyakarta for the cooperation in assisting the development of cocoa cluster in Nglangeran village.

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